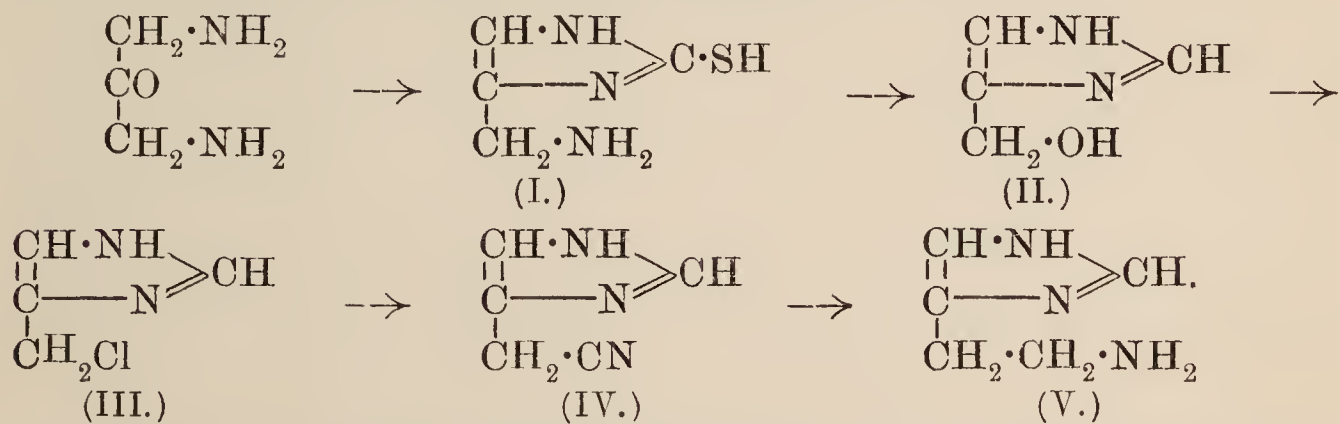


***90. "A new synthesis of 4(or 5)- β -aminoethylglyoxaline, one of the active principles of ergot." By Frank Lee Pyman.**

The following synthesis of 4(or 5)- β -aminoethylglyoxaline affords a convenient method for its preparation.

Diaminoacetone dihydrochloride when heated with potassium thiocyanate yields 2-*thiol*-4(or 5)-*aminomethylglyoxaline* (I) [m. p. 188° (corr.)], which on oxidation with nitric acid gives 4(or 5)-*hydroxymethylglyoxaline* (II) [m. p. 93—94° (corr.)]. The hydrochloride of the latter is converted by phosphorus pentachloride into 4(or 5)-*chloromethylglyoxaline hydrochloride* (III) [m. p. 144—145° (corr.)], and this, when suitably treated with potassium cyanide, gives rise to 4(or 5)-*cyanomethylglyoxaline* (IV) [m. p. 138—140° (corr.)]. The latter base, on reduction with sodium and alcohol, yields 4(or 5)- β -aminoethylglyoxaline (V).



A number of salts of these compounds, and several derivatives obtained as by-products in the various stages of the synthesis, were also described.

***91. "The synthesis of *r*-histidine." (Preliminary note.)
By Frank Lee Pyman.**

4(or 5)-Chloromethylglyoxaline hydrochloride, of which the synthetical preparation is described in the preceding communication, is a valuable compound for the synthesis of substances containing the glyoxaline complex, for it reacts readily with ethyl sodioacetoacetate, ethyl sodiomalonate, and similarly constituted compounds, forming the corresponding 4(or 5)-glyoxalinemethyl ($\text{C}_3\text{H}_3\text{N}_2\cdot\text{CH}_2^-$) derivatives. By the use of this salt, the synthesis of *r*-histidine has been effected as follows. 4(or 5)-Chloromethylglyoxaline hydrochloride and ethyl sodiochloromalonate readily condense, yielding *ethyl* 4(or 5)-*glyoxalinemethylchloromalonate*

(I), of which the *sesquioxalate*, $(C_{11}H_{15}O_4N_2Cl)_4(C_2H_2O_4)_3$, melts and decomposes at 176° (corr.). This base, on hydrolysis with 20 per cent. hydrochloric acid, gives *r-α-chloro-β-glyoxaline-4(or 5)-propionic acid* (II) [m. p. 201° (corr.)], which, when heated with strong ammonia at 110° , yields *r-histidine* (*r-α-amino-β-glyoxaline-4(or 5)-propionic acid*, (III) [melting and decomposing at 283° (corr.)], identical in all respects with that obtained by racemising the naturally-occurring amino-acid *l-histidine*.

